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(54) Sintered magnesium-based composite material and process for preparing same.

(57) A magnesium-based composite material having an improved mechanical strength, particularly the modulus of elasticity thereof, with a relatively low density is provided by pressing and sintering a mixture of magnesium or magnesium-based alloy particle or a metal particle mixture of magnesium with other metal(s) with a reinforcement that may be of boron, or boron-coated BC_4, Si_3N_4, SiC, Al_2O_3 or MgO.

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Sintered Magnesium-Based Composition Material and Process for Preparing Same

The present invention relates to a sintered magnesium-based composite material and a process for preparing the same.

Magnesium alloys have attracted attention as a light-weight, high mechanical strength, metal. They are used in aircraft and space equipment and components and in electronics equipment and components.

In the field of electronics equipment and components, mechanical parts for magnetic recording, particularly a head arm, often comprise a diecast article made of a magnesium alloy. The important characteristics of the material for a head arm include low density and high mechanical strength, particularly the Young's modulus of elasticity. Magnesium and magnesium-based alloys are good candidates for such a head arm due to their low density, but they have a low Young's modulus of elasticity.

It would therefore be desirable to be able to provide a magnesium, or magnesium-based, alloy material that has increased modulus of elasticity without significant increase in density. If a head arm were made of such a material it would be possible to obtain an improvement in the performance of a magnetic recording as a result of an increase in the speed of movement of the head.

A method of improving the modulus of elasticity of a magnesium alloy is known, in which a very small amount of zirconium or a rare earth metal is added to prevent a growth of the crystal grains of the magnesium, but this provides only a low modulus of elasticity of about 4500kgf mm^{-2}.

In Japanese Unexamined Patent Publication (Kokai) No.55-161495 published on December 16, 1980, H.Inoue et al., disclose a vibrating plate for a sonic converter, comprising a fused alloy of magnesium and boron. A fused or cast alloy fo magnesium and boron, however, does not provide a uniform composition due to the difference of the densities of the magnesium and teh boron, and therefore, does not provide the expected improved properties.

Sintering magnesium powders in the form of a shape to obtain a sintered body of that shape is known, but do not provide a body having a sufficient Young's modulus of elasticity.

A sintered material according to the invention has a matrix of magnesium or a magnesium-based alloy and is characterised in that it includes reinforcement dispersed in the matrix. The reinforcement that is used, and the amount of the reinforcement, is selected in order that the sintered material has the desired properties, and in particular generally in order that the modulus of elasticity of the material is substantially greater than it would be in the absence of the reinforcement, although the density is not significantly increased. The reinforcement should be distributed substantially uniformly throughout the matrix and the material is normally the product obtained by sintering a compress formed of particles of the magnesium or magnesium-based alloy and that has the reinforcement substantially uniformly distributed throughout.

The reinforcement is normally a material that is added to the magnesium or magnesium-based alloy, and the preferred added materials are boron or boron-coated materials selected from boron carbide, silicon nitride, silicon carbide, aluminium oxide and magnesium oxide. Another suitable reinforcement is magnesium oxide formed by oxidation within the matrix.

As explained in more detail below, the matrix may be magnesium or a magnesium-based alloy that is formed mainly of magnesium, for instance being formed of at least 88% magnesium. Magnesium aluminium alloys are particularly suitable.

The preferred materials of the invention are the materials that have a reinforcement comprising boron or a boron-coated material selected from boron carbide, silicon nitride, silicon carbide, aluminium oxide and magnesium oxide. The properties of the relevant materials are shown in Table 1, which also shows the properties of magnesium.

Table 1

Material	Density (g/cc)	Modulus of elasticity (kgf/mm^2)
Magnesium	1.74	4.5×10^3
Boron	2.55	4.0×10^4
Boron carbide	2.52	4.6×10^4
Silicon nitride	3.10	3.5×10^4
Silicon carbide	3.12	5.0×10^4
Aluminium oxide	3.99	3.7×10^4
Magnesium oxide	3.65	2.5×10^4

Boron is the most preferable of the materials shown in Table 1, since boron does not easily react with magnesium and does not mechanically weaken a composite. Conversely boron carbide, silicon nitride, silicon carbide, aluminium oxide, and magnesium oxide react with magnesium to form a mechanically weak composite produce, and as a result, mechanically weaken the composite or cause deficiencies therein. Nevertheless, boron carbide (B_4C), silicon nitride, silicon carbide, aluminium oxide, and magnesium oxide may be used as a reinforcement for magnesium, without the above-mentioned problems, if the surface of the silicon nitride, etc., is coated with boron.

Accordingly, the reinforcement used in the present invention can be selected from the group of boron and boron-coated, boron carbide, silicon nitride, silicon carbide, aluminium oxide, and magnesium oxide, and this reinforcement may be in any form, for example, powder, whiskers, and short fibres. The size of the reinforcement is not partiuclarly limited, but preferably, the maximum size of the reiforcement is 0.1μm to 1mm, more preferably 0.1μm to 100μm. Up to 50% by volume of the reinforcement can be dispersed in the matrix of magnesium or magnesium alloy, which is obtained by sintering magnesium or magnesium alloy powder. The amount of reinforcement is preferably in the range 2 to 30% by volume. Preferably the amount is 2 to 25%, most preferably 4 to 25%, but best improvement in mechanical strength while maintaining satisfactory density is generally obtained with amounts of from 4 to 20% by volume.

The coating of the reinforcement such as silicon nitride, etc., with boron can be carried out by any suitable method, although a gas phase deposition method such as CVD, sputtering, or evaporation is most convenient. As described above, boron is most preferable from the viewpoint of the inert nature thereof with magnesium, but boron is a relatively expensive material and, therefore, a boron-coated material such as silicon nitride or the like provides an advantage of a lower cost.

The matrix of magnesium or magnesium-based alloy is not particularly limited, in that a magnesium-aluminium system (particularly 3-12 wt% Al), a magnesium-aluminium-zinc system (particularly 3-9 wt% Al and 0.1-3.0 wt% zinc), and a magnesium-zirconium-zinc system may be used as this magnesium-based alloy.

The magnesium-based composite of the present invention is prepared by sintering a mixture of magnesium particles and reinforcement. Sintering is advantageous in that it provides a uniform dispersion of the boron-based reinforcement in the matrix by forming a mixture of magnesium particles and a reinforcement into a shape close to the desired final shape and allows a uniform dispersion of the boron-based reinforcement in the matrix in the final sintered shaped product.

Accordingly, in another aspect of the present invention, there is provided a process for preparing a sintered magnesium-based composite material, comprising the steps of: preparing a mixture of magnesium or magnesium-based alloy particle or a mixture of magnesium particle with other metal particle(s) with a reinforcement selected from the group of boron and boron-coated boron carbide, silicon nitride, silicon carbide, aluminum oxide and magnesium oxide, the reinforcement being in an amount of 2 to 30% by volume of the mixture; pressing the mixture at a pressure of 1 to 8 tons/cm^2 to form a shaped body; and heating the shaped body at a temperature of 550 to 650°C in an inert atmosphere to obtain a sintered magnesium-based composite material. The sintered magnesium-based composite material may be further subjected to an HIP treatment to increase the density thereof.

The magnesium or magnesium-based alloy or a metal mixture of magnesium with other metal(s) may have a particle size of 0.1 to 100 μm. The magnesium-based mixture is a mixture of magnesium with another metal or metals by which a magnesium-based alloy is formed by the following sintering process.

The pressing may be carried out in the conventional manner.

The sintering of the shaped body is carried out in an inert atmosphere, for example, under an argon or helium gas flow of 1 to 10 ℓ/min, at a temperature of 550 to 650 °C, for 10 minutes to 10 hours or more. A relative density of 95 to 98% may be obtained by this sintering process. For the sample sintered at about 600 °C, which exhibites the highest modulus of elasticity, the structure is relatively dense and necking among the particles occurs. However, when sintered at 550 °C, the structure is less dense. At 650 °C, the structure is too coarse to be strengthened.

In a further aspect of the present invention, there is provided a process for preparing a sintered magnesium-base composite material, comprising the steps of: pressing a magnesium-based particle to form a porous magnesium-based body; heating the porous shaped body in an oxidizing atmosphere to form a sintered magnesium-based body containing magnesium oxide therein; and subjecting the sintered magnesium body to a plastic deformation processing to increase a relative density of the sintered magnesium-based body due to a reinforcement by the magnesium oxide.

In this process, the sintered magnesium-based body containing magnesium oxide therein is subjected to a plastic deformation process to increase the relative density thereof, and as a result, the magnesium matrix and magnesium oxide are made into a composite without heating or a reaction therebetween, i.e., without mechanically weakening the composite.

The starting magnesium-based particle may be a particle of magnesium, a magnesium alloy, or a mixture of magnesium and another metal or metals forming a magnesium alloy. The above particle typically has a size of 1 to 100 μm.

The pressing is carried out at a pressure of 0.5 to 4 tons/cm^2 to form a porous body having a relative density of 50% to 93%, and the sintering is carried out at a temperature of 500 to 600 °C in an oxidizing atmosphere, for example, an argon atmosphere containing 50 to 1000 ppm of oxygen, for 10 minutes to 10 hours.

The plastic deformation of the sintered body may be carried out by, for example, pressing, rolling swagging, etc.; for example, it may be pressed at a pressure of 1 to 8 tons/cm^2.

According to the present invention, a magnesium-based material has an improved mechanical strength, particularly the modulus of elasticity thereof, and no substantial loss of the small density thereof, as shown in the following Examples. The sintered magnesium-based composite material according to the present invention has an additional advantage in that the thermal expansion coefficient of the magnesium-based material can be adjusted by an appropriate selection of the composition of the composite. This ability to adjust the thermal expansion coefficient prevents a mismatch of the thermal expansion coefficient of a head arm with a recording disc, so that a deviation of the head from the tracks formed on a disc of e.g., aluminum, can be prevented.

The present invention will now be described by way of Examples, and with reference to the drawings in which:

Figure 1 shows the relationship between the density of the Mg-B composite and the amount of boron added:

Figure 2 shows the relationship between the modulus of elasticity of the Mg-B composite and the amount of boron added;

Figure 3 shows the relationship between the tensil strength of the Mg-B composite and the amount of boron added;

Figure 4 shows the relationship between the thermal expansion coefficient of the Mg-B composite and the amount of boron added;

Figure 5 shows the dependence of the modulus of elasticity on the aluminium content; and

Figures 6A and 6B show the results of XMA analysis for samples containing 6, and 9 percent Al by weight and 10 percent B by volume.

Example 1

A powder mixture of Mg-9 wt% Al was prepared by mixing a -200 mesh magnesium powder and -325 mesh aluminium powder, and a boron powder (average particle size of 20μm) was mixed with the above powder mixture in an amount of 0 to 30% by volume.

The resultant powder mixture was pressed at 4 tons/cm^2 to form a tensile sample test piece, and the sample test piece was sintered in an argon atmosphere at 560-620 °C for 1 hour.

The density, the modulus of elasticity (Young's modulus), the tensile strength, and the thermal expansion coefficient of the resultant sintered body was evaluated, and the results were as shown in Figs. 1

to 4.

In Figs. 1 to 4, the density of the composite material was 1.8 g/cm^3 at most, which is almost the same as the 1.83 g/cm^3 of the density of a conventional magnesium alloy for a head arm (AZ91: a magnesium alloy with 9 wt% Al and 1 wt% Zn). On the other hand, the modulus of elasticity was improved to 6300 kgf.mm^2, 1.4 times larger than that of the conventional magnesium alloy (AZ91), and the tensile strength was 20 kgf/mm^2, about 2 times larger than that of the conventional magnesium alloy (AZ91). It can be seen that 2 to 30% by volume of boron is preferable from the viewpoint of increasing the modulus of elasticity. and that the thermal expansion coefficient was varied or decreased as the amount of added boron was increased. Namely, an addition of about 6 to 7.5% by volume of boron provided a composite having a thermal expansion coefficient equivalent to that of an aluminum alloy generally used for a magnetic recording disc substrate.

To determine the dependence of the modulus of elasticity on the Al content, the Al content of the B Mg sintered composite system was varied.

To determine the optimum composition, the aluminum content was varied between 0 and 18 wt%. to determine the composition dependency of the modulus of elasticity.

The dependence of the modulus of elasticity on aluminum content is shown in Fig. 5. The modulus of elasticity has a value of 6300 kgf.mm^2 (1.4 times higher than that of the cast Mg-Al alloy without boron) when the aluminum content is 9% by weight. In comparison, without boron, the optimum aluminum content is 6% by weight.

Figures 6A and 6B show the results of XMA analysis for the samples containing 6, and 9 percent Al by weight, and 10 percent B by volume. Both samples have a uniform distribution of Al and Mg in the matrix. However, the sample containing 9% Al by weight has an aluminum-rich layer several microns in thickness around the boron particles. This concentration of aluminum around the boron particles may promote good boron-magnesium interface bonding, resulting in a B/Mg-Al alloy with high modulus of elasticity. This aluminum concentration may explain the differences in the optimum aluminum content for the samples with or without boron.

A magnesium-aluminum sintered alloy, reinforced with boron particles has been developed that has an increased modulus of elasticity. Light weight magnesium-aluminum alloys have proved to be viable candidates for high-speed moving components used in computer peripherals. To improve the modulus of elasticity, a composite material technique has been used in which boron particles reinforce the alloy matrix.

Sintering in argon or helium near the temperature of 600 °C is optimum for the magnesium-aluminum alloy, since no brittle phases are found.

XMA analysis revealed that an aluminum-rich interface layer which forms around the boron particles may promote the formation of strong bonds between the boron particulate reinforcement and the magnesium-aluminum matrix.

Example 2

Powders of boron carbide, aluminum oxide, silicon nitride and silicon carbide, having a particle size of about 1-50 μm, were charged in a respective chemical vapor deposition apparatuse, and using boron chloride (BCl_3) and hydrogen as the reaction gases and a temperature of 800 to 1000 °C, the following chemical reaction was caused for 10 minutes, to obtain a coating of boron on the above particles, the coating having a thickness of 1 to 3 μm:

$$2BCl_3 + 3H_2 \rightarrow 2B + 6HCl$$

The coated powders were mixed with a -200 mesh magnesium alloy (Mg-9 wt% Al) in an amount of 10% by volume of the coated powders based on the total volume of the mixture. The obtained mixtures of powders were pressed at 4 tons/cm^2 and sintered in an argon atmosphere at 600 °C for 1 hour.

The densities, the moduli of elasticity, and the tensile strengths of the resultant samples were then evaluated, and the results were shown in Table 2.

Table 2

Reinforcing Material	Density ($g.cm^3$)	Modulus of Elasticity ($kgf.mm^2$)	Tensile strength ($kgf.mm^2$)
SiC		6500	25.3
B_4C		6400	24.1
Al_2O_3		6200	24.7
Si_3N_4		6000	21.8
B *		6300	22.5
Mg **	1.69	3800	8.0

* Data from a composite using 10 vol% of boron powder.
** Data from Mg-9% Al alloy.

Example 3

A -200 mesh magnesium powder was pressed at 2 tons/cm^2 to form a porous magnesium shaped body having a relative density of 85%.

The porous magnesium body was heat treated in a gas flow of argon containing 200 ppm of oxygen, at 500 °C for 1 hour, and a sintered magnesium body containing a thickness of 0.1 to 2 μm of magnesium oxide inside pores of the body, a relative density of the sintered body being 87%, was obtained.

This sintered magnesium body containing magnesium oxide was pressed again at 4 tons/cm^2 to obtain a shaped body of a Mg-MgO composite. This composite shaped body had a relative density of 96%, and the properties shown in Table 3.

Table 3

Reinforcing Material	Density ($g.cm^3$)	Modulus of Elasticity ($kgf.mm^2$)	Tensile strength ($kgf\ mm^2$)
Mg-MgO composite	1.76	5400	11.5
Sintered Mg	1.69	3800	8.0

Claims

1. A sintered material that has a matrix of magnesium or a magnesium-based alloy characterised in that its modulus of elasticity is increased by a reinforcement dispersed in the matrix and that comprises boron, a boron-coated material selected from boron carbide, silicon nitride, silicon carbide, aluminium oxide and magnesium oxide, or magnesium oxide formed by oxidation within the matrix.

2. A material according to claim 1 in which the matrix is a magnesium-based alloy with aluminium.

3. A material according to claim 1 or claim 2 in which the reinforcement comprises boron or a boron-coated material selected from boron carbide, silicon nitride, silicon carbide, aluminium oxide and magnesium oxide.

4. A material according to claim 3 in which the reinforcement comprises boron.

5. A material according to claim 3 in which the reinforcement comprises boron-coated silicon nitride, silicon carbide and aluminium oxide.

6. A material according to any of claims 3 to 5 in which the reinforcement is in the form of powder, whiskers or short fibres.

7. A material according to any of claims 3 to 5 in which the reinforcement has a maximum dimension of 0.1μm to 1mm, preferably 0.1 to 100μm.

8. A material according to any of claims 3 to 7 in which the amount of the reinforcement is from 2 to 30%, preferably 4 to 25%, by volume.

9. A material according to any preceding claim that has the reinforcement substantially uniformly distributed throughout the matrix and that has been made by sintering a compress that has the reinforcement substantially uniformly distributed throughout and that is formed of particles of magnesium, particles of magnesium-based alloy, or particles of magnesium and particles of alloying metal.

10. A process for forming a material according to any of claims 3 to 8 comprising

preparing a mixture of magnesium or magnesium-based alloy particle or a mixture of magnesium particle with another metal particle with a reinforcement selected from the group of boron and boron-coated boron carbide, silicon nitride, silicon carbide, aluminium oxide and magnesium oxide, the reinforcement being in an amount of 2 to 30% by volume of the mixture;

pressing said mixture at a pressure of 1 to 8 tons/cm^2 to form a shaped body: and

heating the shaped body at a temperature of 550 to 650°C in an inert atmosphere to obtain a sintered magnesium-based composite material.

11. A process according to claim 10, further comprising the step of subjecting said sintered magnesium-based composite material to an HIP treatment.

12. A process for preparing a sintered magnesium-based composite material, comprising the steps of:

pressing a magnesium-based particle to form a porous magnesium-based body;

heating the porous shaped body in an oxidising atmosphere to form a sintered magnesium-based body containing magnesium oxide therein: and

subjecting the sintered magnesium body to a plastic deformation process to increase a relative density of the sintered magnesium-based body by a reinforcement of magnesium oxide.

Fig. 1

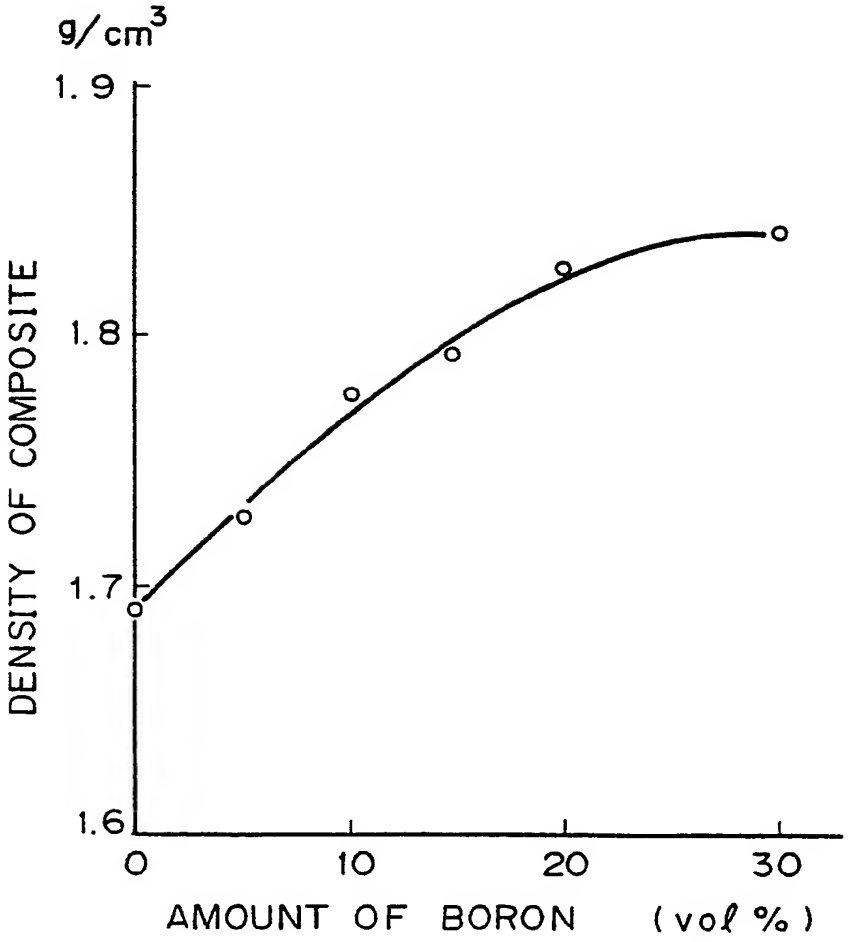


Fig. 2

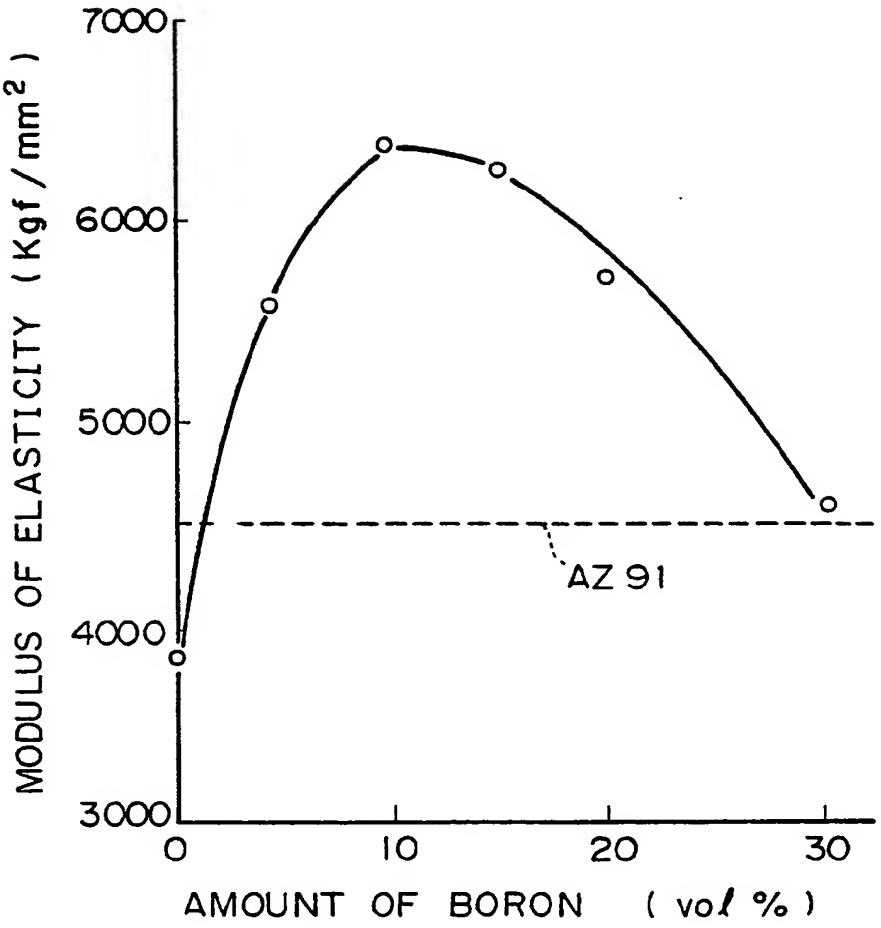


Fig. 3

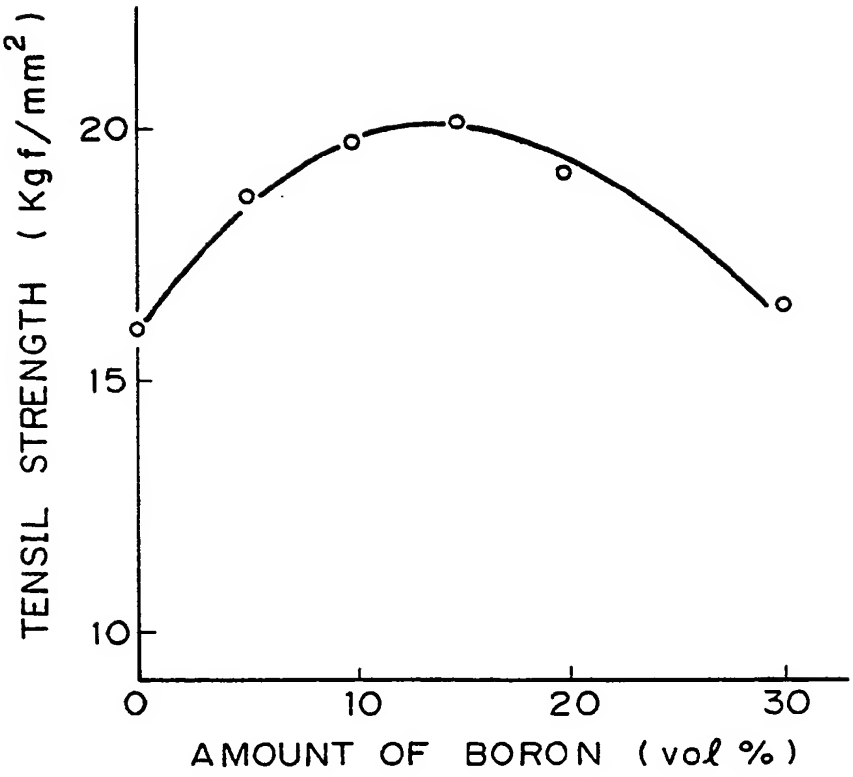


Fig. 4

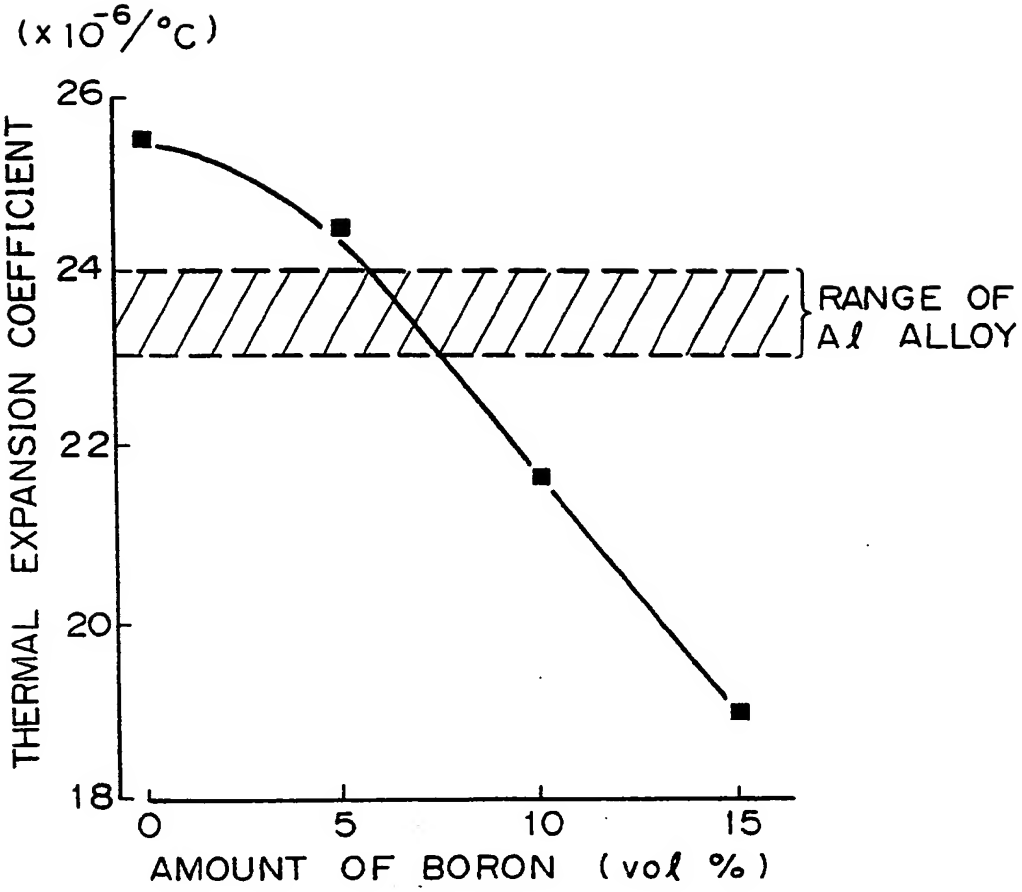
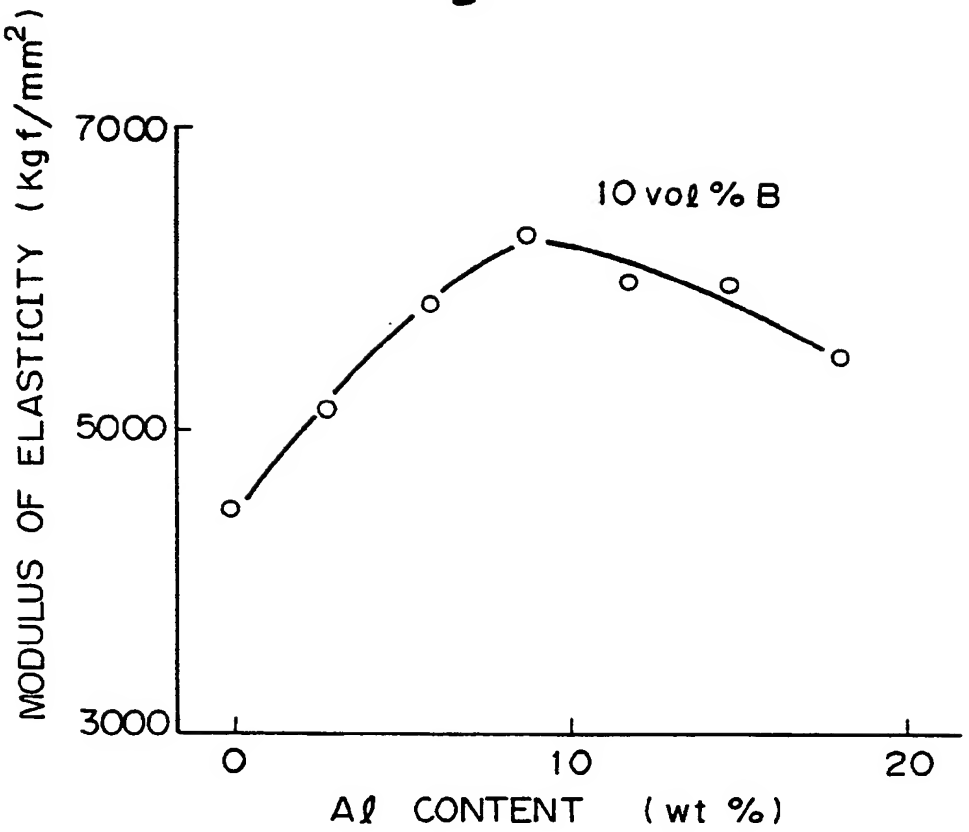

(x 10⁻⁶/°C)
26
24
22
20
18
THERMAL EXPANSION COEFFICIENT
RANGE OF Al ALLOY
0
5
10
15
AMOUNT OF BORON (vol %)

Fig. 5



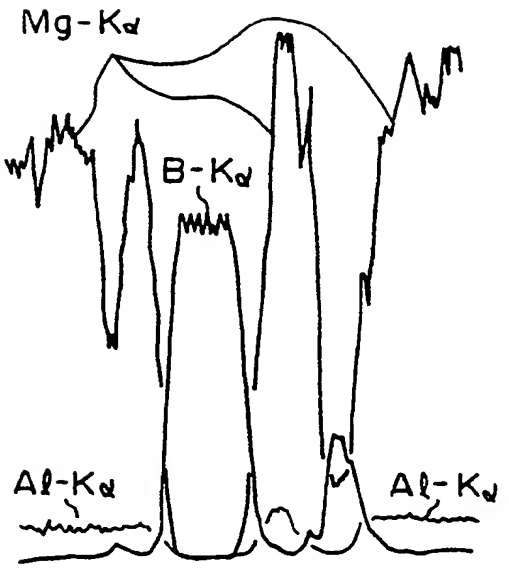


Fig. 6A

6 wt % Al

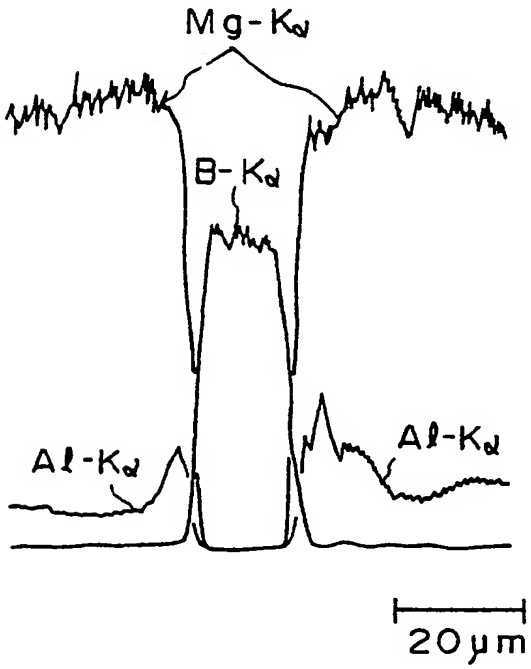


Fig. 6B

9 wt % Al

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Bundesdruckerei Berlin

European Patent Office

EUROPEAN SEARCH REPORT

Application number

EP 88 31 1738

DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int Cl 4)
X	EP-A-0 240 251 (BRITISH PETROLEUM) * Claims 1-13 * --	1-11	C 22 C 32/00 C 22 C 1/10 C 22 C 1/09
X	US-A-3 216 824 (J. BOGHEN et al.) * Claims 1-3; column 2, line 70 - column 3, line 11; column 4, examples 1,2 * --	1,12	
A	US-A-3 775 530 (G.D. LAWRENCE et al) * Claims 1,6,8,9 * --	3-5,10	
A	DE-A-2 657 685 (RES. INST. FOR IRON, STEEL AND OTHER METALS OF THE TOKOKU UNIV.) * Claims 1,6,8 * --	3-5,10	
A	PATENT ABSTRACTS OF JAPAN, vol. 7, no. 209 (C-186), September 14, 1983 & JP-A-58 107 435 (NIPPON DENSO K.K.) 27-06-1983 * Abstract * --	3-5,10	TECHNICAL FIELDS SEARCHED (Int Cl 4) C 22 C
A	PATENT ABSTRACTS OF JAPAN, vol. 10, no. 128, C-345, May 13, 1986 & JP-A-60 251 247 (KOGYO GIJUTSUIN) 11-12-1985 * Abstract * ----------		

The present search report has been drawn up for all claims

Place of search	Date of completion of the search	Examiner
THE HAGUE	03-10-1989	SCHRUERS

CATEGORY OF CITED DOCUMENTS

X : particularly relevant if taken alone
Y : particularly relevant if combined with another document of the same category
A : technological background
O : non-written disclosure
P : intermediate document

T : theory or principle underlying the invention
E : earlier patent document, but published on, or after the filing date
D : document cited in the application
L : document cited for other reasons

& : member of the same patent family, corresponding document

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European Patent Office

CLAIMS INCURRING FEES

The present European patent application comprised at the time of filing more than ten claims.

☐ All claims fees have been paid within the prescribed time limit. The present European search report has been drawn up for all claims.

☐ Only part of the claims fees have been paid within the prescribed time limit. The present European search report has been drawn up for the first ten claims and for those claims for which claims fees have been paid.

namely claims:

☐ No claims fees have been paid within the prescribed time limit. The present European search report has been drawn up for the first ten claims.

X LACK OF UNITY OF INVENTION

The Search Division considers that the present European patent application does not comply with the requirement of unity of invention and relates to several inventions or groups of inventions.

namely:

1. Claims 2-11 and 1 partially:
   Mg compound produced by dispersion of a reinforcement in the alloy and its process of production

2. Claims 12 and 1 partially:
   Mg compound produced by internal oxidation and its process of production

☒ All further search fees have been paid within the fixed time limit. The present European search report has been drawn up for all claims.

☐ Only part of the further search fees have been paid within the fixed time limit. The present European search report has been drawn up for those parts of the European patent application which relate to the inventions in respect of which search fees have been paid,

namely claims:

☐ None of the further search fees has been paid within the fixed time limit. The present European search report has been drawn up for those parts of the European patent application which relate to the invention first mentioned in the claims.

namely claims: